# International Standard 5373

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION●МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ●ORGANISATION INTERNATIONALE DE NORMALISATION

# Condensed phosphates for industrial use (including foodstuffs) — Determination of calcium content — Flame atomic absorption spectrometric method

*Phosphates condensés à usage industriel (y compris les industries alimentaires) — Dosage du calcium — Méthode par spectrométrie d'absorption atomique dans la flamme*

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# Foreword

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International Standard ISO 5375 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in July 1980.

It has been approved by the member bodies of the following countries :

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No member body expressed disapproval of the document.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

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**INTERNATIONAL STANDARD** **ISO 5373-1981 (E)**

# Condensed phosphates for industrial use (including foodstuffs) — Determination of calcium content — Flame atomic absorption spectrometric method

## 1 Scope

This International Standard specifies a flame atomic absorption spectrometric method for the determination of the calcium content of condensed phosphates for industrial use (including foodstuffs).

## 2 Field of application

The method is applicable to products having calcium contents equal to or greater than 50 mg/kg.

The presence of soluble silica ($SiO_2$) up to 5 µg/ml in the test solution does not interfere.

## 3 Principle

Preliminary hydrolysis by boiling a test portion in the presence of hydrochloric acid. Addition of sodium ions, until a constant concentration is obtained, to stabilize the promotion and emission of calcium, and addition of lanthanum chloride to increase the sensitivity of the method. Aspiration of the solution into a dinitrogen monoxide-acetylene flame and determination of the calcium content by spectrometric measurement of the absorption of the 422,7 nm line emitted by a hollow-cathode calcium lamp.

## 4 Reagents

During the analysis, use only reagents of recognized analytical grade and only water doubly distilled in borosilicate glass apparatus with ground glass joints, or water of equivalent purity.

**4.1 Lanthanum chloride heptahydrate** ($LaCl_3.7H_2O$), 100 g/l solution.

**4.2 Hydrochloric acid,** solution containing approximately 220 g of HCl per litre.

**4.3 Phosphoric acid,** 40 g/l solution, free from calcium, corresponding to 29 g of $P_2O_5$ per litre.

Weigh, to the nearest 0,1 g, 29 g of phosphorus(V) oxide ($P_2O_5$) and spread out in a shallow layer in a suitable dish. Allow the dish to stand in a closed vessel containing water (for example a desiccator containing water in place of the desiccant), in order to effect the initial hydration. Then dissolve the hydrated phosphorus(V) oxide in 1 000 ml of water.

NOTE — If a grade of orthophosphoric acid containing less than 5 mg of Ca per kilogram is available commercially, it can be used instead of phosphorus(V) oxide to prepare this solution by diluting 44 g of 90 % $H_3PO_4$ solution ($\rho$ approximately 1,75 g/ml), or its equivalent, to 1 000 ml with water.

**4.4 Sodium chloride,** 255 g/l solution.

1 ml of this solution contains approximately 100 mg of $Na^+$.

**4.5 Calcium,** standard solution corresponding to 1,000 g of $Ca^{2+}$ per litre.

Weigh, to the nearest 0,000 1 g, 2,497 2 g of calcium carbonate, previously dried at about 250 °C for 2 h and cooled in a desiccator. Place it in a beaker of suitable capacity (for example 600 ml) and dissolve carefully in 30 ml of the hydrochloric acid solution (4.2). Dilute the solution and transfer quantitatively to a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

1 ml of this standard solution contains 1,000 mg of $Ca^{2+}$.

Store this solution in a bottle of material free from calcium.

**4.6. Calcium,** standard solution corresponding to 0,050 g of $Ca^{2+}$ per litre.

Transfer 50,0 ml of the standard calcium solution (4.5) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 50 µg of $Ca^{2+}$.

Prepare this solution just before use.

## 5 Apparatus

Ordinary laboratory apparatus, of material free from calcium, and

**5.1 Atomic absorption spectrometer,** equipped with a burner designed to operate with acetylene and dinitrogen monoxide.

**5.2 Hollow-cathode calcium lamp.**

## 6 Procedure

**WARNING — The use of the dinitrogen monoxide-acetylene flame presents some hazards. Handle the gases with suitable precautions and operate the apparatus under an efficiently ventilated fume hood.**

### 6.1 Test portion

Weigh, to the nearest 0,000 2 g, about 5 g of the test sample.

### 6.2 Preparation of the calibration graph

#### 6.2.1 Preparation of standard matching solutions

Into each of a series of five 100 ml one-mark volumetric flasks, place a quantity of the phosphoric acid solution (4.3) such that its $P_2O_5$ concentration is the same as that of the test solution (6.3.1) (see for example annex A).

Add 5 ml of the sodium chloride solution (4.4) and 5 ml of the hydrochloric acid solution (4.2) and then, respectively, the volumes of the standard calcium solution (4.6) shown in table 1.

**Table 1**

| Standard calcium solution (4.6) | Corresponding mass of Ca |
|---|---|
| ml | µg |
| 0* | 0 |
| 1,0 | 50 |
| 2,0 | 100 |
| 4,0 | 200 |
| 6,0 | 300 |

* Blank test on reagents for calibration.

To each flask, add 1 ml of the lanthanum chloride solution (4.1), dilute to the mark and mix.

NOTE — The total quantity of calcium contained in the reagents used for the preparation of the standard matching solutions [except the standard calcium solution (4.6)], should be lower than 50 µg.

#### 6.2.2 Adjustment of the apparatus

Fit the hollow-cathode calcium lamp (5.2) in the apparatus (5.1), switch on the current and allow to stabilize. Adjust the current, the sensitivity and the aperture of the slit according to the characteristics of the apparatus. Adjust the wavelength in the region of 422,7 nm to maximum absorbance. Adjust the pressure of the acetylene and of the dinitrogen monoxide according to the characteristics of the aspirator/burner. Adjust the aspiration rate to between 2 and 4 ml/min.

#### 6.2.3 Spectrometric measurements

Aspirate the series of standard matching solutions (6.2.1) in succession into the flame and measure the absorbance for each. Take care to keep the aspiration rate constant throughout the preparation of the calibration graph. Aspirate water through the burner after each measurement.

#### 6.2.4 Plotting the graph

Plot a graph having, for example, the masses, in micrograms, of $Ca^{2+}$ contained in 100 ml of the standard matching solutions as abscissae, and the corresponding values of the measured absorbance, corrected for the value of the absorbance measured in the blank test on reagents for calibration (6.2.1 — zero term), as ordinates.

### 6.3 Determination

#### 6.3.1 Preparation of the test solution

Transfer the test portion (6.1) to a beaker of suitable capacity (for example 600 ml) and dissolve it in 250 ml of water and 25 ml of the hydrochloric acid solution (4.2). Cover the beaker with a clock glass and boil for 20 min. Cool the solution and transfer it quantitatively to a 500 ml one-mark volumetric flask. Add 5 ml of the lanthanum chloride solution (4.1) and the amount of sodium chloride solution (4.4) required to obtain a sodium concentration of approximately 5 mg/ml in the final solution, including the sodium contained in the test portion (6.1) (see for example annex A which refers to pure products). Dilute to the mark and mix.

If the calcium content is between 50 and 200 mg/kg, carry out the measurements directly on the test solution thus obtained.

If the calcium content is higher, carry out further dilutions as indicated in table 2.

**Table 2**

| Expected Ca content | Aliquot portion of the test solution (6.3.1) to be taken | Hydrochloric acid solution (4.2) to be added | Sodium chloride solution (4.4) to be added | Lanthanum chloride solution (4.1) to be added | Final volume of the solution |
|---|---|---|---|---|---|
| mg/kg | ml | ml | ml | ml | ml |
| 200 to 500 | 50 | 2,50 | 2,50 | 0,50 | 100 |
| 500 to 1 000 | 25 | 3,75 | 3,75 | 0,75 | 100 |
| 1 000 to 1 500 | 20 | 4,00 | 4,00 | 0,80 | 100 |
| 1 500 to 2 000 | 10 | 4,50 | 4,50 | 0,90 | 100 |

#### 6.3.2 Spectrometric measurements

**6.3.2.1** Preliminary measurement

Carry out a preliminary measurement on the test solution (6.3.1) following the procedure specified in 6.2.3, at the same time as the spectrometric measurements are carried out on the standard matching solutions (6.2.1).

From the calibration graph (6.2.4), calculate the approximate concentration of $Ca^{2+}$ in 100 ml of the test solution (6.3.1).

**6.3.2.2** Bracketing measurement

Carry out a second measurement on the test solution (6.3.1) following the procedure specified in 6.2.3, by bracketing between two standard matching solutions of composition similar to that of the standard matching solutions (6.2.1), but having $Ca^{2+}$ contents which differ by 25 µg/100 ml.

To prepare these standard matching solutions, follow the procedure specified in 6.2.1, using, however, suitable quantities of the standard calcium solution (4.6).

## 7 Expression of results

### 7.1 Concentration of the test solution

The calcium concentration, $c$, expressed in micrograms of $Ca^{2+}$ per 100 ml of the test solution, is given by the formula

$$c = c_1 + (c_2 - c_1)\frac{A_0 - A_1}{A_2 - A_1}$$

where

$c_1$ is the concentration, in micrograms per 100 ml, of the standard matching solution of lower concentration, used for the bracketing measurement (6.3.2.2);

$A_1$ is the corresponding value of absorbance;

$c_2$ is the concentration, in micrograms per 100 ml, of the standard matching solution of higher concentration, used for the bracketing measurement (6.3.2.2);

$A_2$ is the corresponding value of absorbance;

$A_0$ is the value of absorbance corresponding to the test solution (6.3.1).

### 7.2 Content of the sample

The calcium (Ca) content, expressed in milligrams per kilogram, is given by the formula

$$\frac{c}{m} \times \frac{500}{100} \times r_D$$

$$= \frac{c}{m} \times 5 \times r_D$$

where

$c$ is the calcium concentration, expressed in micrograms of $Ca^{2+}$ per 100 ml of the test solution;

$m$ is the mass, in grams, of the test portion (6.1);

$r_D$ is the dilution ratio (see table 2);

5 is the ratio of the volume of the test solution (6.3.1) to the volume of the standard matching solutions (6.2.1).

## 8 Test report

The test report shall include the following particulars :

a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard, or regarded as optional.

# Annex A

## Examples of the addition of sodium chloride solution (4.4) and phosphoric acid solution (4.3) (see 6.2.1 and 6.3.1)

| Product | Formula | Percentage by mass of | | Mass, in grams, in 5 g of test portion (6.1) of | | Volume, in millilitres of the sodium chloride solution (4.4) to be added to the test solution (6.3.1) | Volume, in millilitres, of the phosphoric acid solution (4.3) to be added to the standard matching solutions (6.2.1) |
|---|---|---|---|---|---|---|---|
| | | $P_2O_5$ | Na | $P_2O_5$ | Na | | |
| Pentasodium triphosphate | $Na_5P_3O_{10}$ | 57,9 | 31,2 | 2,90 | 1,56 | 9,4 | 20,0 |
| Tetrasodium diphosphate | $Na_4P_2O_7$ | 53,4 | 34,6 | 2,67 | 1,73 | 7,7 | 18,4 |
| Disodium dihydrogen diphosphate | $Na_2H_2P_2O_7$ | 63,9 | 20,7 | 3,20 | 1,04 | 14,6 | 22,1 |
| Sodium metaphosphates | $(NaPO_3)_n$ | 69,6 | 22,5 | 3,48 | 1,12 | 13,8 | 24,0 |

# Annex B

## ISO Publications relating to condensed phosphates for industrial use (including foodstuffs)

ISO 5372 — Determination of arsenic content — Silver diethyldithiocarbamate photometric method.

ISO 5373 — Determination of calcium content — Flame atomic absorption spectrometric method.

ISO 5374 — Determination of chloride content — Potentiometric method.

ISO 5375 — Determination of oxides of nitrogen content — 3,4-Xylenol spectrophotometric method.